Journal of Molecular Catalysis A: Chemical 147 (1999) 37–40
www.elsevier.com/locate/molcata

The synthesis of sucrose ester and selection of its catalyst

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Abstract

Sucrose fatty acid ester (called sucrose ester for short) is a kind of non-ionic surface active agent with excellent performance and many uses. This paper describes the process of synthesis and selection of its catalyst. The results show that neutral lithium soap oil acid is the best catalyst for the synthesis of sucrose ester. Using mixed alkali metal soap with proper ingredient ratio as catalyst can improve the yield of sucrose ester. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Sucrose ester; Non-ionic surface active agent; Alkali soap; Mixed catalyst; Ester exchange

1. Introduction

Sucrose esters are a type of organic compound, which are formed by combining sucrose with various acid groups. Sucrose esters are excellent non-ionic active surface agents. They are not only used in the industries of detergents, cosmetics and medicine, but also in the area of food. Sucrose esters have very wide arbitrary hydrophilicity and lipophilicity balance (HLB), excellent physical properties, surface activity, no stimulus and environmental pollution, and they are safe to the human body, so they have very wide uses and flexibility, and they are receiving more and more attention in the world [1]. But in China, the research and production of sucrose ester is still in the elementary stage. This experiment is aimed at investigating the best reaction condition, especially selection of the catalyst.

2. Experiment

2.1. Material used to synthesize sucrose ester

The principal materials used in the synthesis of sucrose ester are sucrose and fatty acid ester. The experiment adopted commercial white granulated sugar with high purity as material for producing sucrose ester. In order to accelerate the reaction, white granulated sugar should be dried and ground before it can be used. Natural fatty acid should be used. If sucrose ester is used as food additive, the fatty acid should be saturated and have long chain. This decision is made from stability against oxidizing and taste. Natural grease is the source of fatty acid, which can be obtained from natural grease through alcoholysis reaction under the existence of basic catalyst (usually sodium formate). The catalysts

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PII: S1381-1169(99)00125-9

used in the experiment are neutral alkali soaps and their mixture.

2.2. Synthesis of sucrose ester

Ester exchange reaction is used to synthesize sucrose ester. The reaction can be expressed with following formula:

$$ROH + R'COOR'' \Leftrightarrow R'COOR + R''OH$$

Because this reaction is reversible, it will no longer move forward when it reaches equilibrium state. In order to get more ester, R″OH, one of the products, must be removed from the system. The reaction velocity is very slow at room temperature and without catalyst. Increasing temperature will accelerate the reaction. If temperature is too high, the solvent will evaporate and the product is decomposed. So the temperature should be controlled within a certain range. Fusion method is used to synthesize sucrose ester [2–4].

2.3. Experimental steps [5]

Powdered white granulated sugar, and methyl carbitylpalmitate (mole ratio of them is 2:1) was used, with a neutral alkali soap catalyst which accounts for 25% of the weight of white granulated sugar. Reaction bottle which can be stirred with magnetic bar was placed in oil bath of 185°C which can be heated. After the reaction temperature reached 185°C, the reactant was stirred for 14 min. And then the vessel was moved into the second oil-bath of 170°C and continually stirred for 10 min under pressure of 133.32 Pa to 399.96 Pa. The reaction was stopped by inflating helium gas and then methylcarbitylcitrate was added into the vessel. 1.1 mole citric acid was added in every mole of metal soap. The reaction products were dissolved in normal butanol and washed with 5% salt water. The normal butanol was removed by heating product in vacuum and extraction with dry nitrogen gas. The remains were extracted with chloroform and analyzed with thin film chromatographic process.

2.4. Composition analysis

The composition was analyzed with column chromatographic method (CC) [6] using silica gel as adsorbent and chloroform as solvent. Sucrose ester was separated into monoester, diester, triester and polyester.

3. Results and discussion

3.1. Experiment using neutral soap as catalyst

Using neutral soap catalyst could ensure the completion of the reaction within 30 min at temperatures of 170°C to 185°C. However, using other catalysts will need several hours to complete the reaction. The mole ratio of white granulated sugar to methylcarbityl palmitate is 1.0:0.8. In the three experiments, the weight percent of sodium oleate relative to white granulated sugar is 10%, 20%, 30%, respectively. By analyzing the chloroform extraction solution of the reaction mixtures we obtained the following data.

3.2. Selection of the catalyst

The experiment showed that using neutral alkali soap is better. But not all the alkali soaps were appropriate. Here, we still use the former reaction as an example to study the performance of each alkali soap. The catalysts used in control test were simple metal soaps or mixed soaps. The mole ratio of white granulated sugar to methylcarbityl palmitate was 1:1. The data is shown in Table 2.

Table 1 shows that the yield of sucrose ester is highest when the weight percent of sodium oleate relative to white granulated sugar is 20%. When sodium oleate accounts for 30% of white granulated sugar, though the total yield of su-

Table 1
Data of experiment using sodium oleate as catalyst

Reaction product		Weight percent of sodium oleate (relative to white granulated sugar)		
		10%	20%	30%
Weight percent of compatible sucrose ester in chloroform		15.56	35.36	21.17
Composition of sucrose ester (% weight)	Monoester	24.39	25.50	35.28
	Diester	42.33	40.21	41.84
	Triester and polyester	16.66	17.01	6.38

crose ester was less than that of sucrose ester above, the content of monoester was higher. If the amount of sodium oleate were over 40%, it would not be good for yield and economy. But had sodium oleate not been used, sucrose ester would have not been produced [7,8].

Data in Table 2 show that, except for lithium palmitate, all other soaps and mixed soaps listed in the table are good catalysts for ester exchange reaction. Of the simple soaps, lithium oleate is the best catalyst, but 94.5% of its product is sucrose ester over triester. By comparing experiment 5 with experiment 7, it can be seen that adding 2% of lithium oleate could improve the catalytic activity of potassium palmitate. The situation of experiment 3 and 6 is similar to above. The experiment also show that lithium palmitate alone has no catalytic activity because lithium palmitate does not dissolve in the reaction mixture. The experiment also indicated that sodium, lithium and potassium salts of short chain fatty acids such as butanoic acid, hexoic acid have very small catalytic activity.

When soda soap and potash soap is used as catalyst, in order to get sucrose ester containing more monoester, the mole ratio of white granulated sugar to fatty acid ester should be 1:1. If the ratio is decreased, the content of polyester in the products will increase. In addition, using lithium soap of unsaturated fatty acid as catalyst, even though the ratio of feed stock is 1:1, the content of polyester in the products is very high. The reason is not clear [1].

Table 2
Performance of catalysts

Number	Catalysts		Yield of sucrose ester (% weight)	Composition of sucrose esters (% weight)		
	Type	Weight (relative to 100 g white granulated sugar)		Monoester	Diester	Triester and polyester
1	Lithium oleate	25.50	37.92	1.45	3.80	94.50
2	Lithium palmitate	25.50	0	0	0	0
3	Sodium oleate	25.50	21.17	39.43	46.65	9.38
4	Sodium palmitate	25.50	9.78	49.02	32.11	18.25
5	Potassium palmitate	25.50	10.03	29.07	34.30	34.97
6	Sodium oleate + Lithium oleate	25.50 + 5	41.40	33.25	35.91	28.56
7	Potassium palmitate + Lithium oleate	25.50 + 2	14.45	34.96	29.55	27.93
8	Potassium palmitate + Lithium oleate	25.50 + 5	16.15	34.39	26.79	29.40

4. Conclusion

(1) Adopting neutral soap as catalyst to synthesize sucrose ester will shorten ester exchange time 2–3 h relative to other method and the yield of production is high; (2) if sodium oleate was used as catalyst, reaction temperature should be kept between 190°C and 170°C. When sodium oleate accounts for 20% of the weight of white granulated sugar, the yield of sucrose ester is higher; (3) when neutral soaps were used as catalysts, if only one catalyst was used, lithium oleate was best catalyst, and if mixed soap was used, mixture of lithium oleate and sodium oleate with proper composition ratio can improve the yield of sucrose ester.

Experiment also indicates that C_{12} fatty acid, which is the main composition of copra oil and palm nut oil, is the best raw material for producing sucrose ester with high water solubility.

When synthesizing sucrose ester, if we not only use mixed catalyst such as mixture of sodium oleate and lithium oleate, but also choose pure natural matter as food stock such as hydro-cotton-seed oil, glycerin monoester, glycerin diester, the remnant alcohol, a byproduct, will do no harm to human when the sucrose ester is used in the industry of detergent, cosmetics and medicine.

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